

DESIGN AND CONSTRUCTION OF MASS
SPECTROGRAPH COMPONENTS

by

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INTRODUCTION

The realization of relatively accurate determinations of the masses of atoms has been accomplished by the use of the mass spectrograph. The mass spectrograph development dates as far back as 1897 when Thomson (7) performed his now classical experiment to determine the charge to mass ratio of the electron. Later, in 1910, Thomson modified his apparatus to measure the charge to mass ratio of a singly charged atom. The first element analyzed was neon of average atomic weight 20.2 which was shown to consist of two isotopes of atomic mass numbers 20 and 22. Since then many modifications in design have been made so that the modern spectrograph is now an instrument of precision.

There are four major fields of application for which the mass spectrograph is now used. These fields employ the mass spectrograph or a mass spectrometer¹ for accurate isotope mass determination, for analysis of chemical mixtures by identification of isotopic masses, to determine relative abundances of different isotopes, and to separate isotopes of a given element in quantity.

Spectrographs designed for accurate isotope mass determinations emphasize high resolving power, with the consequent reduction of large quantity separation. On the other hand, spectrographs used for quantity separation relax the high resolving power. When used for analysis, both high resolving power and capability for large quantity separation are de-emphasized in the interests of simplicity and versatility.

¹The mass spectrometer differs from the mass spectrograph only in the collection and detection of the isotopes. The mass spectrograph uses a photographic plate detector while the spectrometer employs some sensitive electrical means for detection.

The construction of the spectrograph, designed by Lewis and Hayden (2), was carried out by More (3). The spectrograph was constructed as a tool for isotope analysis mainly in the rare-earth region, and as a small quantity separator of radioactive materials. These requirements demanded an instrument somewhat between that of an analytical and a precision type spectrograph.

The purpose of the research reported here was to develop the basic instrument into an operating tool capable of application to the fields for which it was initially designed.

THEORY OF THE MASS SPECTROGRAPH

The basic principle underlying the operation of a mass spectrograph is the action of a magnetic field on a moving charged particle.

A particle of mass M , velocity v and charge e , when projected into a uniform magnetic field of strength B , describes a circular trajectory of radius R , according to the well-known formula;

$$\frac{Mv^2}{R} = \frac{Bev}{10} \quad (1)$$

By a simple manipulation this formula reduces to;

$$R = \frac{\sqrt{2ME \times 10^2}}{Be} \quad (2)$$

Here, E , equal to $\frac{1}{2}Mv^2$, is the kinetic energy of the particle.

The mass spectrograph employs an electrostatic field to give a group of ions an energy E . When this ion beam is directed into a uniform magnetic field the ions of different masses describe circular arcs of different radii of curvature, and thus may be separated from each other.

To express the radii of curvature in terms of measurable quantities, the kinetic energy E may be replaced by the work done on the ion by the accelerating potential, V ;

$$E = \frac{1}{2}Mv^2 = Ve \times 10^7 \quad (3)$$

Equations 2. and 3. then yield the following formula;

$$R = \frac{\sqrt{2MV \times 10^9}}{B\sqrt{e}} \quad (4)$$

Making use of the relation;

$$M = \frac{A}{N} \quad (5)$$

where A is the atomic weight and N is Avogadro's number, equations 4. and 5. yield the following formula;

$$R = \frac{\sqrt{2AV \times 10^9}}{B\sqrt{eN}} \quad (6)$$

Substituting the known constants;

$$N = 6.025 \times 10^{23}$$

$$e = 1.602 \times 10^{-19} \text{ coulombs}$$

into equation 6. we obtain;

$$R = 1.4395 \times 10^2 \frac{\sqrt{AV}}{B} \quad (7)$$

In these equations, masses are in grams, distances in centimeters, charge in coulombs, magnetic field in gauss, and potential in practical volts.

DESIGN AND CONSTRUCTION

The High Voltage Power Supply

The output of the high voltage power supply accelerates ions produced in the mass spectrograph to high energies. A continuously variable potential of from 5 to 10 kilovolts was required, and a high order of stability of this potential was demanded to minimize line broadening.

A radio-frequency type of power supply was selected because of the voltage range desired, the greater ease in filtering, and because of the lighter and smaller components required. The output voltage was variable from 5 to 9.5 kv. positive, and determined the available output current, the allowed range of which is shown in Plate I.

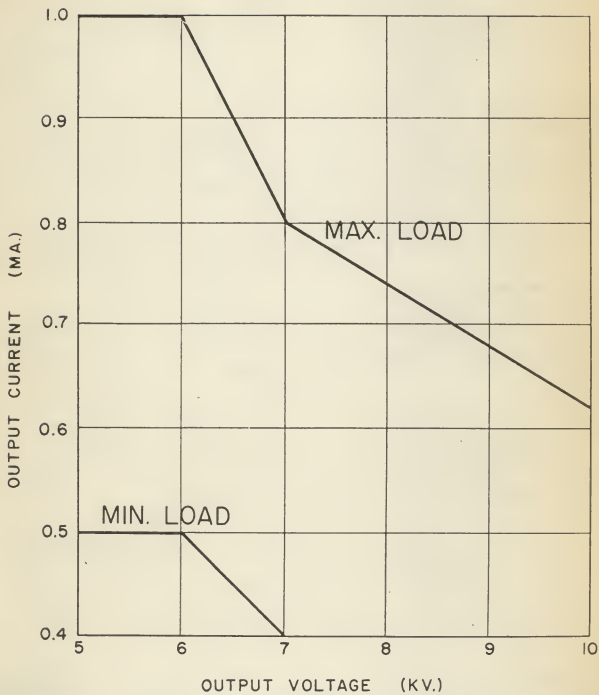
The circuit diagram for the power supply is shown in Plate II. V13 and V14 form, with coil L1, a tuned-plate oscillator operating in the region of 175 kc. The high voltage from the secondary of L1 is rectified by V15 and then filtered by a dual-section resistance-capacitance filter. Ripple content of the output voltage was of the order of 0.0001 percent when the filter components and all wiring carrying high voltage d.c. were carefully shielded from the field of L1. Omission of this shielding resulted in ripple content as high as 5 percent.

Adjustment of the output voltage was obtained by varying the output of V13 and V14. Therefore the usual procedure of obtaining power for the filament of V15 from coil L1 would result in dependence of the temperature of this filament upon output voltage. Since this procedure would shorten the life of the tube, and possibly degrade its operating characteristics, a separate oscillator was constructed to supply the recommended 0.25 watts of

EXPLANATION OF PLATE I

Graph of output current versus output voltage for the high voltage power supply.

PLATE I

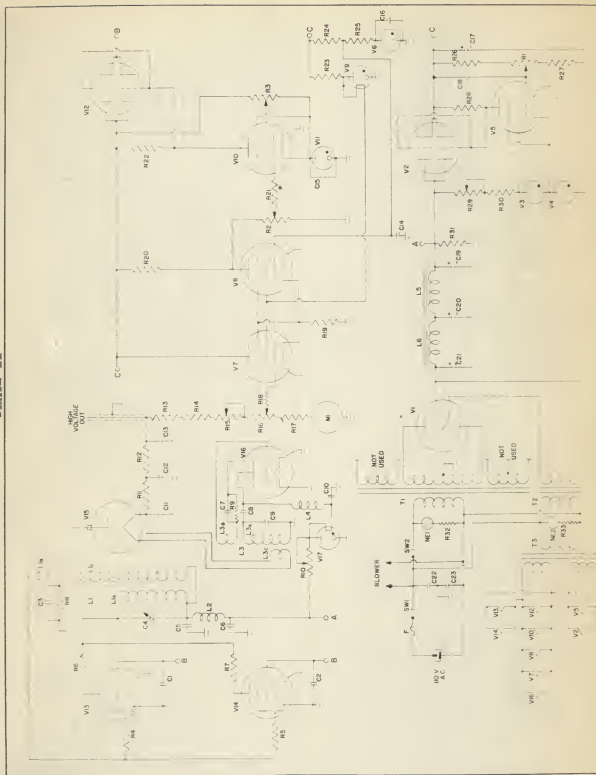


EXPLANATION OF PLATE II

The High Voltage Power Supply

R1 - 150K Ohmite "AB" potentiometer
 R2, R5 - 2.5 meg. Ohmits "AB" potentiometer
 R3 - 100K Ohmite "AB" potentiometer
 R4, R5, R21 - 580 ohm
 R6, R7 - 47 ohm
 R8 - 47K 2 watt
 R9 - 2400 ohm
 R10 - 7500 ohm 10 watt adjustable
 R11, R12 - 100K 1 watt
 R13 - 94 meg. Victoreen RK-3 precision
 R14 - 3 meg. precision
 R16 - 1 meg. Ohmite "AB" potentiometer
 R17 - 0.97 meg. precision
 R18 - 1 meg.
 R19 - 0.2 meg. 1 watt
 R20, R22, R28 - 0.47 meg.
 R23 - 15K 2 watt
 R24 - 75K 1 watt
 R25 - 4.7K
 R26 - 180K
 R27 - 100K
 R29 - 4000 ohm 10 watt adjustable
 R30 - 1500 ohm 2 watt
 R31 - 16K 10 watt
 R32, R33 - 220K
 C1, C2, C3, C5, C6, C7, C8, C10 - 0.01 mfd.
 600 v. mica or ceramic
 C4 - 2900-4555 mfd. adjustable
 C9 - 1000 mfd.
 C11, C12, C13 - 500 mfd. 20 kv.
 C14 - 0.5 mfd. 600 v. oil filled
 C15, C16 - 100 mfd. 200 v. mica or ceramic
 C17 - 20 mfd. 450 v. electrolytic
 C18 - 0.1 mfd. 400 v. paper

C19, C20, C21 - 4 mfd. 600 v. electrolytic
 C22, C23 - 0.001 mfd. 600 v. mica or ceramic
 L1 - Spellman 10-15 kv. high voltage coil
 L1A - 168 microhenry
 L1B - 30 microhenry
 L1C - 4100 microhenry
 L2 - 2.5 mh. 250 ma. R.F.C.
 L3 - Oscillator coil for high voltage rectifier
 filament supply
 L3A - 325 microhenry
 L3B - 19 microhenry
 L3C - 4 turn link
 L4 - 2.5 mh. 125 ma. R.F.C.
 L5, L6 - Thordarson T-20054 d.c. choke. 8 hv. @ 150 ma.,
 4 hv. @ 200 ma.
 T1 - Thordarson T-22R35 power transformer. 800 v.c.t.,
 @ 340 ma., 5 v. @ 6 a., 6.3 v. @ 7 a.
 T2 - 5 v. @ 3 a. filament transformer
 T3 - 6.3 v. @ 2.75 a., 6.3 v. @ 5.5 a. filament trans-
 former
 F - 3AG 5A fuse
 SW1, SW2 - SPST toggle switch
 NE1, NE2 - NE-51 neon lamp
 NI - 0-100 microammeter
 V1 - 5R4GX
 V2, V12 - 6AS7G
 V3, V4 - 6B2
 V5, V7, V8, V10 - 6SU7
 V6, V11 - 5651
 V9 - VR105/OC3
 V13, V14 - 6L6
 V15 - 1B3GT
 V16 - 6J5
 V17 - VR150/OD3



power to the filament of V15. This oscillator, comprised of V16 and L3, is a tuned-plate circuit operating in the region of 275 kc. Since recommended operating parameters for tubes are given at frequencies of 60 cycles or lower, the filament voltage of V15 could not be used to ascertain whether or not the tube was operating at its correct filament temperature. To obtain proper operation of V15, the filament of a 1B3GT was heated from a conventional filament transformer having the recommended 1.25 volt secondary. Optical comparison was then made between the filaments of this tube and V15, and link L3_C was adjusted so that both tubes were operating at the same filament temperature.

The bleeder resistors, R13-17, in the output circuit serve three purposes. First, they discharge the filter capacitors when the supply is turned off. Secondly they form, with M1, a voltmeter, calibrated by R15, which measures the output voltage. The third function of the bleeder resistors is to supply a control voltage to the regulating circuit, and to allow the output voltage to be varied over a wide range by adjustment of R16.

Regulation of the output voltage was obtained by controlling the screen voltage of the main oscillator tubes, V13 and V14. This method has been applied by Reilley, et. al., (5), and by Walker (8) with considerable success. A small portion of the output voltage is sampled from the bleeder and amplified by a difference amplifier comprised of V7 and V8. It is amplified further by V10, a d.c. amplifier, and applied to the grid of the series regulator tube, V12, which controls the screen voltage of the main oscillator tubes. These two cascaded d.c. amplifiers have a total gain of approximately 30,000. The low voltage supply to the regulating circuit is stabilized by V2 and V5, thereby reducing output voltage fluctuations due to output current

and line voltage variations. The regulating circuits described above are conventional and are discussed thoroughly by Sealy (6), p. 113, 309.

To obtain proper operation of the regulating circuits the following experimentally obtained voltages were maintained: Voltage at "C", set by R1, was 250 volts. Screen voltage of V10 was adjusted by R3 to 100 volts, and R2 was set to give a reading of 84 volts at the grid of V10 when the plate voltage of V8 was 150 volts.

Measurement of the regulation of output voltage indicated variations of 0.05 percent resulting from 0.1 milliamperes changes in load current. The current supplied to the spectrograph was of the order of one microampere, which was either outside or barely inside the allowed range of currents shown in Plate I. Precision resistors, external to the power supply, were therefore placed across the output to provide an additional load of 0.4 to 0.6 milliamperes and thus insure operation within the allowed current range. These resistors had very small voltage and temperature coefficients, and presented a stable load to the power supply. Therefore, variations in the current to the spectrograph between 0 and 2 microamperes caused the output voltage to change less than 0.001 percent when the external load was connected.

The high voltage output of the supply was constant to within ± 0.05 percent as line voltage varied from 90 to 130 volts. To obtain this low variation the regulating action of the power supply was enhanced by use of a Raytheon voltage stabilizer between the 115 volt line and the input to the high voltage power supply. The output voltage of the stabilizer was maintained at 115 volts ± 0.5 percent for line voltages between 90 and 130 volts.

Variation of the output voltage with time was first measured with no forced cooling of the power supply components. The drift, tabulated in Table 1,

Table 1. Variation of output voltage with time for the high voltage power supply.*

Without forced cooling		:	With forced cooling	
		:		
Time (min.)	Variation from initial voltage (percent)		Time (min.)	Variation from initial voltage (percent)
0	+0.000		0	+0.000
15	+1.750		15	-0.100
30	+1.715		30	-0.290
45	+1.510		45	-0.303
60	+1.357		60	-0.329
75	+1.093		75	-0.293
90	+0.947		90	-0.390
105	-0.068		105	-0.546
120	-0.186		120	-0.594
135	-0.250		135	-0.606
150	-0.385		150	-0.608

*Drift was measured using a Leeds and Northrup type K2 potentiometer having six place accuracy. The potential measured was of the order of one volt, and was obtained from a resistive voltage divider placed across the output of the high voltage power supply. The voltage divider was composed of precision resistors, and was cooled by forced air to reduce the possibility of thermal drift in the measuring circuit. No variations exceeding those given above were observed over the allowed voltage and current ranges.

was considerably more than could be tolerated. A considerable reduction in drift was obtained by cooling all transformers and tubes, as well as the components in the regulating circuits, by means of forced air blowers. The drift characteristics obtained under these conditions are indicated in Table 1. After a three hour warmup, drift over a five minute period was of the order of ± 0.005 percent. The variation was ± 0.02 percent for 30 minutes, and two hours of operation resulted in a total drift of only ± 0.04 percent. These values could be improved if precision resistors were used exclusively in all regulating circuits.

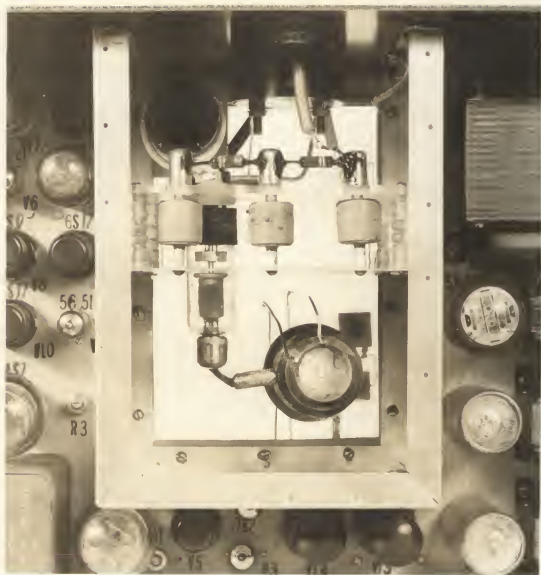
Initial tests conducted on the high voltage power supply indicated that the presence of corona discharges seriously affected the regulation, often causing variations as large as 80 percent in the output voltage. Considerable attention was therefore given to corona preventative devices and techniques during construction. Plate III illustrates the methods used in the high voltage section of the power supply. This section was shielded from the remainder of the supply and was divided into two compartments by an aluminum shield. The lower compartment contained L1 and part of V15, which had potentials of radio-frequency applied to them. The upper compartment contained the components which carried only direct current, which included the filter and bleeder networks. L1 and V15 were sprayed with Krylon acrylic plastic, which is waterproof and has a high dielectric constant. All junction points in both compartments used machined brass terminals or connectors having rounded corners and edges. After final assembly each solder joint was painted with anti-corona lacquer, and the junction points were sprayed with Krylon.

Junction points external to the high voltage power supply were treated with anti-corona lacquer, and then covered either by polyethylene tubing or

EXPLANATION OF PLATE III

The high voltage section of the high voltage power supply.

PLATE III



several wrapped layers of Scotch electrical tape. Both the tubing and tape were judged to be equally effective, and choice between the two was a matter of convenience.

Upon application of the above methods, no corona discharges were observed at potentials up to 15 kv.

The Vacuum Tube Bridge and the Emission-Regulating Circuit for the Ionization Gauge

The vacuum tube bridge serves the dual function of measuring ion beam current and vacuum. Ion beam current was obtained from the nichrome disk of the positive ion accelerator assembly described by Lewis and Hayden (2) and More (3). The vacuum in the spectrograph was measured by means of a type VG-1A ionization gauge, manufactured by Distillation Products, Inc., and accuracy over a wide range of pressures was insured by regulation of the grid current of the ionization gauge by the emission-regulating circuit.

The vacuum tube bridge, the circuit diagram of which is shown in Plate IV, is similar to that employed by Lewis and Hayden (2) and More (3). V7 and V8 form a difference amplifier circuit. With the grid of V8 at ground potential, accomplished by positioning S2 to SET, the null adjust potentiometer, R17, was adjusted so as to zero M2. The current to be measured was then allowed to flow through one of the four different grid resistors, R12-15, which were available through S2 and determined the full scale current sensitivity of the bridge. The non-zero grid potential of V8 then necessitated readjustment of R17 to zero M2. The difference between the two settings of R17, indicated by a dial reading from 0-100, was proportional to the current being measured. Calibration of the dial was obtained

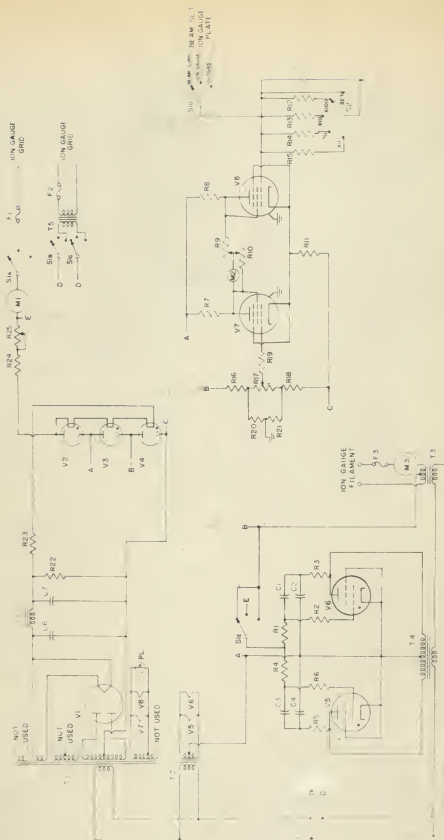
EXPLANATION OF PLATE IV

The vacuum tube bridge and the emission-regulation circuit for the ionization gauge.

R1, R2, R3, R4, R5, R6- 100K
 R7, R8, R11- 47K 1 watt 5%
 R9, R10- 1 meg. dual potentiometer
 R12 - 1K 5%
 R13 - 10K 5%
 R14 - 100K 5%
 R15 - 1 meg. 5%
 R16 - 5.1 K
 R17 - 25K linear taper Ohmite "AB" potentiometer
 R18 - 15K 1 watt
 R19 - 100 ohm
 R20, R21-220 ohm 5%
 R22- 30K 20 watt
 R23 - 5K 10 watt
 C1, C2, C3, C4- 1 mfd. 600 v.
 C5- 0.1 mfd. 100 v.
 C6- 8 mfd. 800 v.
 C7- 8 mfd. 600 v.
 S1- 5 pole 3 position rotary
 S2- 1 pole 5 position rotary
 S3- S.P.S.T. toggle
 M1- 10 ma. d.c. ammeter
 M2- 75 microamp. d.c. galvanometer
 M3- 10 amp. a.c. ammeter

T1- UTC 99900, 1000 v.e.t., @ 100 ma., 6.3 v. @ 8 a.,
 5 v. @ 3 a.
 T2- Stancor P-6134, 6.3 v.e.t., @ 8 a.
 T3- Thordarson T21F18, 10 v.e.t., @ 5 a.
 T4- Thordarson T22R01, 550 v.e.t., @ 50 ma.
 T5- 6.3 v. @ 12 a.
 F1- 1/100 amp. slo-blo
 F2- 10 amp. slo-blo
 F3- 8 amp. slo-blo
 F4- 2 amp.
 FL- #27 pilot lamp
 V1- 5R4GY
 V2- V3- VR150/OD3
 V4- VR105/OC3
 V5, V6- 2051
 V7, V8- 68J7

PLATE IV



by direct measurements of currents supplied to the grid resistors, and is shown in Plate V. The current value from the calibration curve, when multiplied by the scale factor determined by the setting of S2, gave the value of the current being measured. The current range of the bridge, determined by the available grid resistor values, was 0.05 to 1000 micro-amperes. The ability of the bridge to measure currents down to 0.05 micro-amperes enabled pressures as low as 4×10^{-4} microns to be measured with the VG-1A ionization gauge.

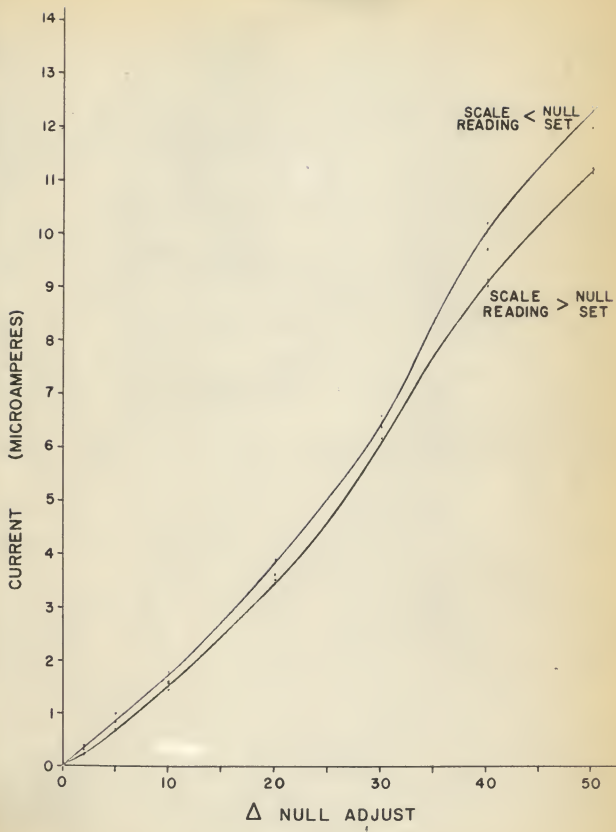
In order to obtain proper operation of the bridge, the static characteristics of V7 and V8 must be closely matched. This condition is insured if, with the grid of V8 at ground potential, the setting of R17 for null is within five percent or less of its electrical midpoint.

The emission-regulating circuit is essentially that described by Nelson and Wing (4). This circuit, by controlling the filament temperature and holding the emission constant, maintained the grid current of the VG-1A at a constant five milliamperes. The diagram of the regulating circuit is shown in Plate IV. V5 and V6 are gas-filled tetrodes having high grid sensitivity. The grid current of the ionization gauge flowing through R24 and R25 produce a voltage drop opposite to the potential across the voltage regulator tube, V2. The resultant potential is applied as a bias voltage to the grids of the gas tetrodes, controlling the firing point of these tubes and the impedance of T4. Thereby, the voltage applied to the ionization gauge filament transformer, T3, is controlled so as to provide constant emission. In order to utilize the full control range of the gas tetrodes, a phase shifting circuit superimposes on the d.c. bias of the grids an alternating voltage which lags the plate voltage by about 90 degrees. When S1 is in the "beam current" or "outgas" position the gas tetrodes are biased

EXPLANATION OF PLATE V

Calibration curve of the vacuum tube bridge.

PLATE V



completely off. The ionization gauge filament then runs too cold to emit, but hot enough to prevent excessive contamination which might produce a burst of gas when the filament is reheated.

The Magnet Current Regulator

The circuit described by Lewis and Hayden (2) and constructed by More (3) was tested to obtain regulation with changing load resistance and drift characteristics. For output currents up to 370 milliamperes, less than 0.01 percent variations in output current occurred for load resistances from 70 to 900 ohms. Output current increased with decreasing load resistance. When the output current was in the 370-470 milliampere range, regulation characteristics were identical, but the load resistances were restricted to values between 400 and 800 ohms. Drift characteristics are indicated in Table 2. After a three hour warmup time, drift over a five minute period was of the order of ± 0.008 percent. The variation was ± 0.03 percent for 30 minutes. These values are about four times those obtained by Lewis and Hayden (2). Forced cooling and the use of precision resistors in all regulating circuits should reduce these figures by a factor of five to ten.

Table 2. Variation of output current with time for the magnet current regulator.*

Time (min.)	:	Variation from initial current (percent)
0		0.000
15		0.092
30		0.124
45		0.147
60		0.164
75		0.179
90		0.192
105		0.202
120		0.218
135		0.233
150		0.248

*Drift was measured using a Leeds and Northrup type K2 potentiometer having six place accuracy. The potential measured was of the order of one volt, and was obtained from a portion of the resistor in the regulator from which the control voltage is obtained.

The Detector

Plates VII and VIII show photographs of the film holder. Three films or plates may be loaded in the holder. When mounted on the spectrograph, each film, or plate, is exposed separately. Therefore, exposures may be made under three different sets of operating parameters. Also, provision was made for operation of the spectrograph with all three films or plates shielded from exposure to the ion beam.

The films or plates were mounted on three sides of a section of square brass, and were held in place by clamps hinged at the bottom of the film mount and secured by a thumbscrew at the top. The film mount was fastened to a brass shaft which passed through its center. At its upper end the shaft was fastened to a brass ring, which bears against the plate at the upper end of the holder and supports the mount-shaft assembly. The shaft

EXPLANATION OF PLATE VII

The assembled detector.

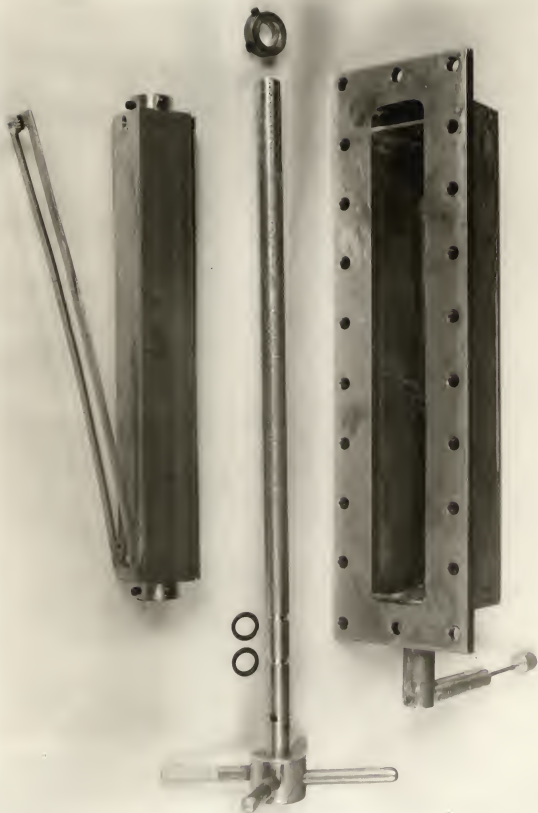
PLATE VII



EXPLANATION OF PLATE VIII

Exploded view of the detector.

PLATE VIII



passes through a brass tube at the bottom of the holder. A vacuum seal was provided by two O-rings positioned between the shaft and the tube. The four spokes on the handle, at the bottom of the shaft, serve to indicate the position of the film mount. Proper positioning of each film or plate with respect to the ion beam was insured by an indexing arrangement, obtained by insertion of a pin into holes drilled into the shaft just above the handle.

This type of detector offers considerable advantage over the conventional type which accommodates only one film or plate. Not only can three exposures be taken without disturbing the vacuum system, but also the operating parameters may be adjusted without having resultant transient effects recorded on the films or plates.

EXPERIMENTAL TECHNIQUES

Preparation of the Ion Source

Positive ions were obtained for analysis in the mass spectrograph by thermal emission from a heated filament. The ion source holder has been described by More (3) and consisted of a tungsten filament, 0.005 inch by 0.030 inch in cross section and 7/16 inch long, and the necessary holder for support and electrical connection.

Prior to deposition of the sample on the filament, the tungsten filament and adjoining surfaces of the holder were cleaned in a dilute solution of nitric and hydrochloric acid which was warmed slightly. Fifty milligrams of each element to be analyzed were then dissolved in one milliliter of dilute nitric acid. Ten microliters of this solution were then pipetted with a small calibrated micropipette onto the filament, and evaporated to

dryness with an infra-red lamp. No difficulty was encountered in obtaining adhesion of the sample to the tungsten filament. This method follows closely that used by Lewis and Hayden (2).

Alignment of the Ion Beam

Careful alignment of the ion beam was necessary to obtain maximum transmission of ions through the instrument and to prevent scattering of ions. Under optimum conditions the ion beam would pass axially through the brass tube which connects the ion source and accelerator to the magnet. More (3) provided for alignment by mounting the ion accelerator assembly on a square brass plate which was connected to the brass tube by a Sylphon bellows. Brass screws, mounted in each of the four corners of the square brass plate, allow for alignment adjustments.


Initial efforts to measure the degree of adjustment with the photographic detector mounted on the camera failed. Exposure due to the ion beam was undetectable, indicating that the majority of the ions reaching the detector had been scattered prior to reaching the camera. Therefore, to obtain an indication of optimum alignment, the photographic detector was mounted approximately one inch from the edge of the pole gap nearest the camera. The developed image thus obtained was due to the non-focused ion beam, and was of the same shape and size as the pole gap of the focusing magnet. Variations in intensity and definition of this image were then recorded for a range of adjustments of the ion accelerator assembly, centered upon an approximated optimum obtained by micrometer and caliper measurements. Adjustment was then made to obtain maximum intensity of the image near the magnet, indicating maximum direct transmission of the ion beam, and to obtain maximum definition at the edges of this image, indicating negligible scattering of the ion beam.

Upon completion of these adjustments, the line structure due to the discrete masses present in the source was then observed with the photographic detector mounted on the camera, which was in the focal plane of the instrument. Further adjustments were then made to obtain greater line intensity, smaller line width, and less background caused by scattering.

During the course of adjustment non-symmetrical curvature of the lines observed at the camera was found to occur. The ion accelerator and all accessible interior surfaces of the spectrograph were thoroughly cleaned to remove any materials which might act as dielectrics and give rise to non-uniform electrostatic fields when ions striking these surfaces were not immediately admitted into the conducting walls. This treatment removed the offending effect of curvature, and the mass spectrum obtained was composed of straight lines as observed by Lewis and Hayden (2) and previous investigators.

The Photographic Detector

Eastman spectroscopic safety film, type SWR (short wave radiation), was used as the photographic detector. The major portion of the gelatin is removed from this film producing a sensitive detector for the deep ultra-violet, but also making the film extremely susceptible to abrasion. A high degree of care was exercised in handling of the film and in no case was any object allowed to touch the emulsion. The film was developed from two to four minutes in Kodak Developer D-19. Exposures of three to six minutes at ion beam currents of one to two microamperes produced well-defined images with good contrast.



EXPERIMENTAL RESULTS

A mixture of lanthanum, praseodymium and neodymium, each containing less than one percent impurities, was analyzed using the previously described techniques. An accelerating potential of 8 kv. and a magnetic field of approximately 10,000 gauss were used with ion beam currents of 0.5 to 2 microamperes to obtain the spectra shown in Plate IX. Two exposures were taken on the film shown in Fig. 1 at slightly different magnetic fields. A single field exposure, shown in Fig. 2, served to indicate the general structure of the mass spectrum.

To enable mass assignments to be made to the individual lines, the heavily exposed broad line was interpreted as a doublet caused by the oxides of La and Pr. Hayden (1) has found that the ionization efficiencies of La and Pr are the highest of the rare earths, thereby indicating that the most intense lines should be assigned to the oxides of these two elements. The ionization efficiency, obtained by Hayden (1), and the relative abundances of the isotopes of Nd indicated that the intensities of the Nd lines should be of the order of one-fiftieth of the intensities of the La and Pr lines.

As the main purpose of the research reported here was to complete the construction of the spectrograph and its components, no detailed analysis of the mass spectra obtained was attempted. However, the spectra do indicate proper operation of the components described in this thesis and, in particular, the resolution obtained verifies the predicted high degree of stability of the power supplies.

The spectra obtained offer a reasonable interpretation of ion, mass, and isotope assignments for some of the lines. The oxides of the three test elements are assigned as shown in Plate IX; however, the assignments are not

EXPLANATION OF PLATE IX

The mass spectrum of a mixture of lanthanum, praseodymium, and neodymium.

Fig. 1. Two exposures taken at slightly different magnetic fields.

Fig. 2. A single exposure indicating the structure of the mass spectrum.

PLATE IX

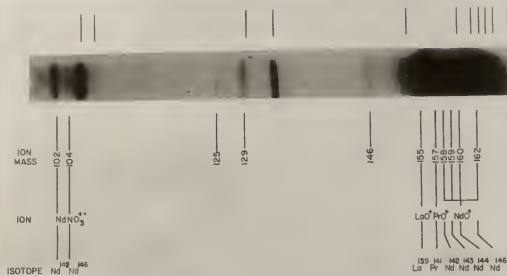


Fig. 1



Fig. 2

definite. The lines at mass numbers 102 and 104 were accounted for by assuming that doubly ionized atoms were formed, although previous results obtained by Hayden (1) indicated no doubly charged ions. The lines at 125, 129, and 146 indicate the presence of impurities, though the high purity of the samples would tend to disallow this. However, the samples had been in stock for some years and the possibility exists that they may have become contaminated. Also, the filament itself could produce ions containing tungsten which would be recorded.

Only one interpretation of the obtained spectra is given here. Final alignment of the ion beam should reduce the line width and resolve the structure more fully. Individual spectra of the La, Pr, and Nd samples, as well as of the filament alone, should then permit valid interpretation of the present results.

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DESIGN AND CONSTRUCTION OF MASS
SPECTROGRAPH COMPONENTS

by

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A mass spectrograph is an instrument which is capable of separating isotopes according to their masses. The operation depends on the fact that a moving charged particle has a circular path in a magnetic field where the radius of curvature, for singly charged ions, is given by;

$$R = 1.4395 \times 10^2 \frac{AV}{B}$$

Here, A is the atomic weight of the particle, V is the potential in volts through which the particle is accelerated prior to entry into the magnetic field, and B is the magnetic field in gauss.

It was the purpose of the present thesis to develop a previously constructed spectrograph into an operating tool capable of isotope analysis mainly in the rare earth region and small quantity separation of radioactive materials. For this study a high voltage power supply was constructed to provide the ion accelerating potential, an emission regulating circuit for the ionization gauge and vacuum tube bridge was constructed to provide for measurement of high vacuum and ion beam current, a detector was constructed which permitted more efficient utilization of the spectrograph, and finally, regulation and drift characteristics of the previously constructed magnet current regulator were measured to insure stable operation of the instrument.

The major problem encountered in this construction was that of the high voltage power supply. It had to be well regulated for changes in load and in line voltages. High voltage was obtained from a radio-frequency oscillator and a step-up coil. For regulation a simple feedback system was used which sampled a portion of the high voltage and corrected any changes in the output voltage by control of the screen potential of the radio-frequency oscillator through proper amplifying circuits. Caution had to be exercised in preventing

corona discharges and other losses due to high voltage. The final result was a regulated supply furnishing from 5 to 9.5 kilovolts with a high degree of stability.

The vacuum tube bridge was capable of measuring currents in the range of 0.05 to 1000 microamperes and, when used with a VG-1A ionization gauge, enabled pressures as low as 4×10^{-4} microns to be measured. The emission regulating circuit, by maintaining the grid current of the ionization gauge at a constant value, allowed vacuum measurements to be made with a high order of accuracy and stability.

The detector was constructed to accommodate three photographic films or plates and provided for separate exposure of each to the ion beam, thereby allowing exposures to be made under three different sets of operating parameters without disturbing the vacuum system. Also, provision was made for operation of the spectrograph with all three films or plates shielded from the ion beam, thus allowing adjustment of the operating parameters without having resultant transient effects recorded on the films or plates.

The samples were handled in the form of nitrates in a dilute nitric acid solution, which were deposited on a tungsten filament, from which ions were obtained by thermal emission. Upon analysis of a test sample of lanthanum, praseodymium and neodymium, spectral lines were obtained but sufficient exposures were not made to enable the lines to be given definite mass assignments.

